Future Developments

There are two areas in porphyrin separation which are expected to develop further in the future. The first is in column technology. The improvement achieved by the introduction of base-deactivated reversed-phase is expected to continue and better columns with improved resolution and reproducibility can be expected. The second is in online LC–MS–MS operation. With the introduction of high sensitivity and resolution mass spectrometers, analysis of porphyrins will be a lot easier in the future.

See also: **II/Chromatography: Liquid:** Detectors: Mass Spectrometry.

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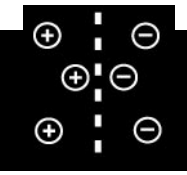
POWDERED RESINS: CONTINUOUS ION EXCHANGE



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Introduction

The use of finely divided (powdered) forms of ion exchange resins began in the early 1960s. Prior to that, synthetic ion exchange resins were manufactured as granules, or preferably spherical beads. The ion exchange granules and beads were used in packed beds to treat liquids, most commonly water. The Graver Water Conditioning Company pioneered the use of the powdered ion exchange resins made by grinding ion exchange resin beads or granules into powders. They discovered that a thin layer of powdered resin offered a dramatic improvement in ion exchange reaction rate versus a conventional packed bed of resin. In 1966 Joseph A. Levendusky of Graver Water Conditioning patented a powdered resin system (named Powdex®) that has been the basis for most practical applications of this technology. This process utilized a pre-coat of powdered ion exchange resins applied to a septum or filter. Thus, the pre-coat process incorporated ion exchange and filtration into one unit operation.

Basic Principles

The original pre-coats were made by combining powdered anion exchange resin with powdered cation exchange resin in a water slurry. Both types of resin were ground moist (40–60% moisture content) into powders using grinding equipment such as hammer mills. This grinding process resulted in a distribution of particle sizes, typically from 1 to 200 μm in diameter. These distributions were centred in the 35–70 μm range. Thus, the powdered ion exchange particles are roughly two orders of magnitude smaller than conventional ion exchange resins (**Figure 1**). The grinding process also results in a tremendous increase in surface area and, consequently, a higher surface area to weight ratio.

Particle size of powdered ion exchange resins is the most important factor in determining performance of pre-coats. In these applications, resin particle size influences ion exchange capacity utilization, filtration efficiency, filtration capacity and pre-coat characteristics (integrity, uniformity, lifetime). In their classic paper, Frisch and Kunin elucidated the effects of

Figure 1 Powdered ion exchange resins.

particle size on kinetics (the rate of reaction) and, hence, utilization of capacity for ion exchange resins. Specifically, both exchange rate and utilization are inversely proportional to particle diameter. **Figure 2** illustrates the relationship between particle size and ion exchange kinetics for standard-sized bead, small-sized bead and powdered strongly basic anion resin. Because of their inherently better kinetics, powdered resins typically exhibit equal, or higher, operating capacities to those of bead resin counterparts.

When mixed in water, powdered resin particles of opposite charge flocculate to form a larger, well-defined agglomerate. This agglomerate particle is commonly called a floc. The size of the floc formed

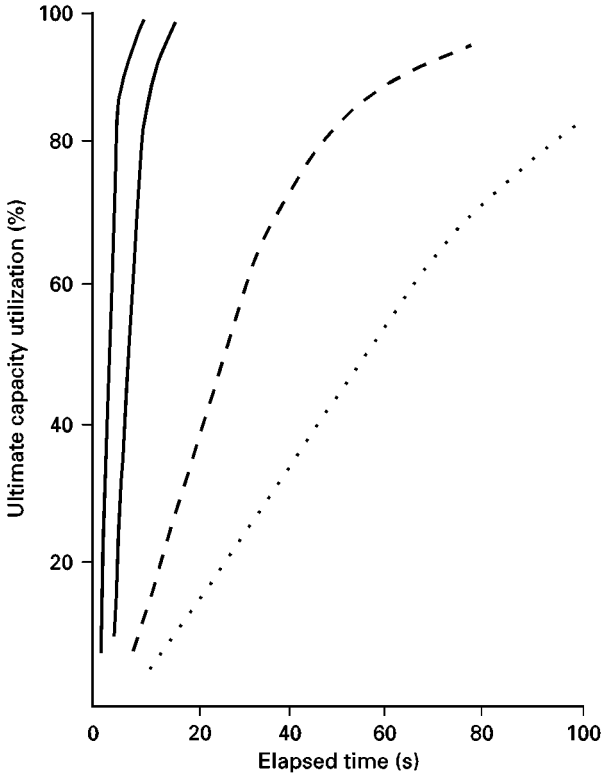

Figure 2 Ion exchange kinetics as a function of particle size. Continuous lines, powdered resin; dashed line, 50–100 mesh; dotted line, 16–40 mesh.

Table 1 Surface charges of powdered resins

Resin type	*Ionic form*	*Zeta potential (mV)*
Strongly acidic cation	Hydrogen	− 42
Strongly acidic cation	Sodium	− 40
Strongly acidic cation	Ammonium	− 30
Strongly basic anion	Hydroxide	+ 48

depends on the surface area and the surface charge of the particles prior to flocculation. The increased surface area resulting from the grinding process explains why powdered resin particles flocculate to a much greater extent than corresponding standard-size resin particles. Electrical charges on the surface of powdered resin particles are measured using zeta potentials. **Table 1** summarizes the zeta potentials for specific ionic forms of common powdered ion exchange resins used in pre-coats.

The flocculated mixture of cation and anion resins has a volume up to 20 times that of the unmixed components in water. As the floc forms, void volume between particles increases dramatically, resulting in this increased bulk volume. Factors, such as the ratio of the two resin components, the ionic forms of the components, the resin slurry concentration, the ionic strength of the solution, the temperature of the slurry and particle sizes of the components and determine the bulk volume of the flocculated mixture.

In addition to their ion exchange capabilities, pre-coats offer excellent filtration. This filtration results from a combination of fine particles (high surface area per unit weight) and significant porosity within the pre-coat. The presence of charged ion exchange sites further enhances the filtration performance. In this regard, the new and freshly regenerated powdered resins found in pre-coats optimize filtration efficiency.

Pre-coat Formulations

The original pre-coat formulations, such as those used in the Powdex® process, were made from just two powdered ion exchange components: a strongly basic anion resin and a strongly acidic cation resin. Typically, these resin components were supplied in the regenerated (ionic) form (H^+ for the cation and OH^- for the anion). These powdered components were added to water and stirred to form a slurry. The bulk volume of the resultant slurry could be controlled by addition of an innocuous organic material, such as dilute polyacrylic acid, which reduces clumping. This organic material binds tightly to the surface of one of the components, typically the anion resin, thus effectively reducing the surface charge of that

component. Use of individual components allows the pre-coat user to adjust the cation to anion ratio according to the requirements of the application. As the technology has evolved, pre-coat manufacturers have offered alternative ionic forms (ammonium, sodium, morpholinium and chloride) and alternative resin chemistry components (weak electrolytes, macroporous exchangers and Type II strongly basic anions) for specific applications.

Premix pre-coats are formulated and tested before shipment to a customer. In contrast to the ultimate flexibility associated with component pre-coats, premixing by the manufacturer guarantees a specific ratio of components and a uniformly mixed material.

In 1976 the first premix incorporating fibre along with the anion and cation resin components was introduced; the corresponding patent was granted 4 years later. Chopped alpha-cellulose fibre was used in this premix and still is the most common fibre constituent in pre-coats. Cellulose fibres are hydrophilic and contain carboxylic groups whose charge facilitates flocculation with resin components. The tendency for fibre flocculation with resin components can be further enhanced by charging the fibre with an ionic polymer with a significant zeta potential. Inclusion of the fibre increases filtration efficiency and minimizes pre-coat cracking caused by the adsorption of colloids on the surface of the anion resin particles. The fibre adds bulk volume and porosity; the amount of bulk depends on the length and rigidity of the fibre: longer and more rigid fibres produce bulky pre-coats and vice versa. A variety of noncellulosic fibres have been used in premixes. Both polyacrylonitrile (PAN) and composite polystyrene/polyethylene fibres are used in current commercial formulations. In the latter case, the polystyrene fibres are functionalized with ion exchange sites, typically sulfonic acid type, to form an ion exchange resin fibre. Fibre can also be incorporated in standard pre-coats via addition as a separate component. Moreover, the fibre can be utilized separately either as an underlay pre-coated prior to the resin components, or conversely, as an overlay pre-coated after the resin components.

Commercial premixes currently popular are available in a variety of component ratios, as shown in **Table 2**. Pre-coats are formulated on a dry weight basis. Many fibres have virtually no moisture content, while most ion exchange resins contain 40–60% water unless dried. The premixes listed in Table 2 are available with either hydrogen form or ammonium form cation resin. The 4 : 5 cation-to-anion ratio common to fibre-containing premixes yields a roughly stoichiometric mix of cation and anion exchange sites.

Table 2 Common premix pre-coat compositions

Resin content (dry weight ratio)		*Mix content (dry weight ratio)*	
Cation resin	*Anion resin*	*Fibre*	*Resin*
1	1	0	1
2	1	0	1
3	1	0	1
4	5	1	1
4	5	1	2
4	5	1	9

Pre-coat Properties

Typically, three properties characterize pre-coats and premixes: moisture content, settling volume and supernate turbidity. Premixes are made from a slurry of the components. The slurry is dewatered by means of centrifugation or a similar mechanical process. The dewatered premix retains water associated with the ion exchange resin sites. Moisture content, or water retention as it is sometimes called, is a measure of the amount of water relative to the solids content of the premix. Typically, moisture contents vary between 50 and 70%. The settling volume measures flocculation of a pre-coat under standardized conditions. The measurements are done in a graduated cylinder with a specific volume and specific concentration of pre-coat slurry. Slurry volume is measured initially and after a specific settling time (t) and calculated according to the following equation:

$$\text{Settling volume } [V/V](\%) = 100 \times (V_t/V_0)$$

Settling volumes typically vary from 40–70%. High settling volumes are indicative of increased pore volume and, consequently, bulky pre-coats, while low settling volumes are indicative of 'tight' pre-coats. As the settling volume increases, depth filtration improves, but ion exchange efficiency tends to decline. Turbidity is measured on the supernatant at the conclusion of the settling volume test. Turbidity is recorded in nephelometric units (NTU): values below 10–15 NTU are considered acceptable. Low turbidity values are indicative of full incorporation of pre-coat components. Typically, anion resins generate more very small particles than cation resins during the grinding process. These fine anion resin particles, in turn, are the least likely to be fully flocculated in the pre-coat and consequently cause turbidity. Fibres are another potential source of turbidity. Fibre particles have weaker charges than resin particles and thus prove harder to flocculate fully in a pre-coat. Fortunately, the addition of the correct amount of the properly charged dilute polyelectrolyte (e.g. polyacrylic acid or polyacrylamide) usually flocculates these

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unincorporated particles and dramatically improves turbidity. Thus, turbidity measurements assist manufacturers in formulating premixes and users in preparing pre-coats satisfactorily from components.

The American Society for Testing and Materials (ASTM) has published validated test methods for measuring properties of the powdered ion exchange resins used in pre-coat materials. Specifically, Standard D 4266-83 covers measurement of the operating capacity for both powdered cation exchange resins (either hydrogen or ammonium form) and powdered anion exchange resins (hydroxide form). Similarly, Standard D 4456-85 encompasses determination of particle size distributions and solids content for powdered ion exchange resins (see Further Reading).

Pre-coat Equipment

Before the introduction of powdered ion exchange resins, materials such as diatomaceous earth and cellulose fibres were coated on to leaf filter elements or wire screen septa. While both these materials and this type of equipment are still used in pre-coat operations, the use of powdered ion exchange resins in pre-coats has led to equipment modifications and improvements.

The basic equipment used for pre-coating with powdered ion exchange resins is fairly simple: a tank for mixing powdered components into a slurry and a filter or septum suitable for retaining the slurry solids. In addition to these basic components, pumps, piping, valves and instrumentation are used in virtually all pre-coat systems. **Figure 3** is a schematic drawing of a typical system used in commercial applications. Vessels used for pre-coating operations generally contain an array of tubular filter elements or septa. These arrays fall into two major designs: top tube sheet and bottom tube sheet. **Figures 4** and **5**, respectively, are schematic drawings of these two designs. The term 'tube sheet' refers to the support structure to which the individual elements are attached. Top tube sheet elements originally were designed to use stainless steel wire cloth or well screen elements. These metal elements have relatively large diameter (0.075–0.150 mm) openings. Bottom tube sheet elements typically use continuously wound yarn elements with much smaller openings (nominal ratings of 0.001–0.010 mm). These filter elements come

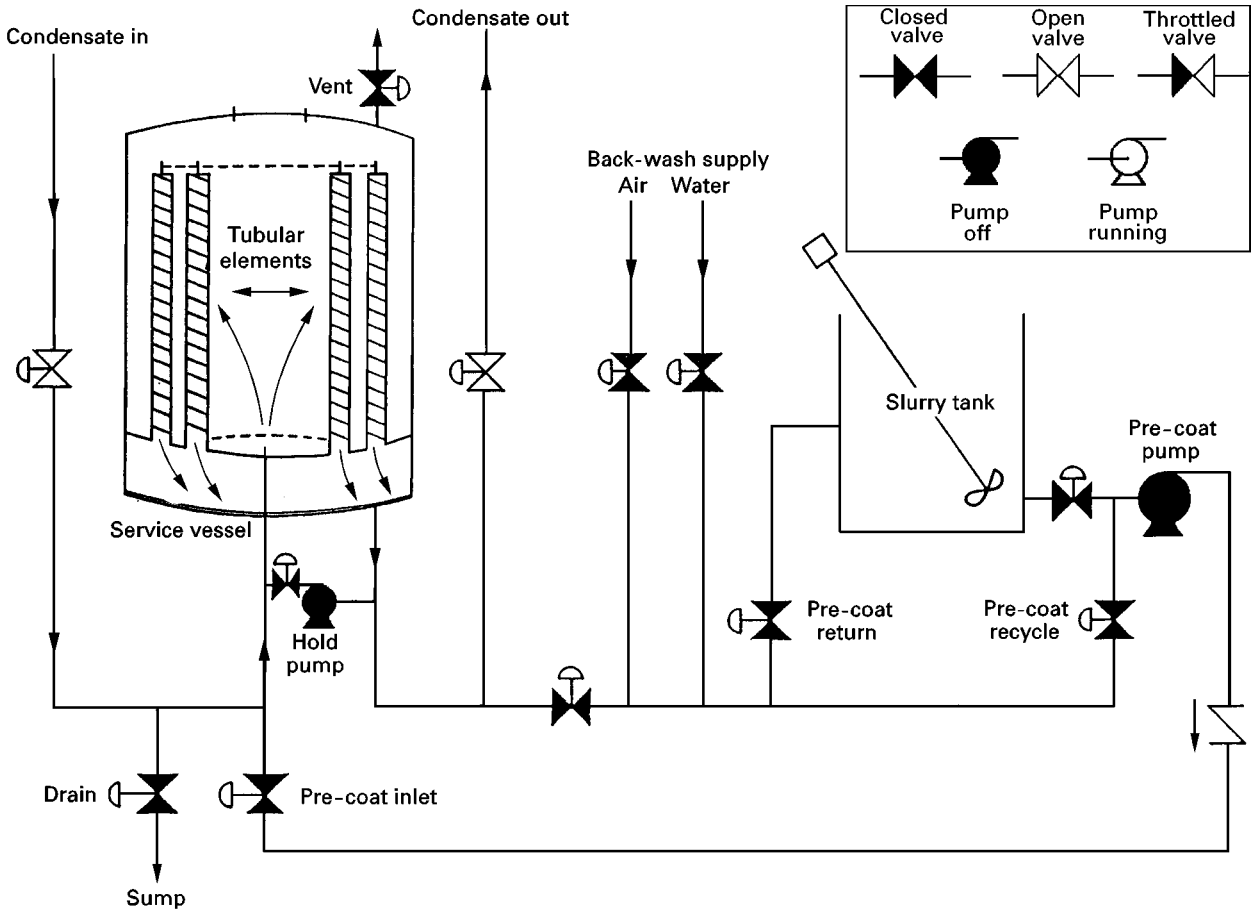

Figure 3 Typical pre-coat system schematic.

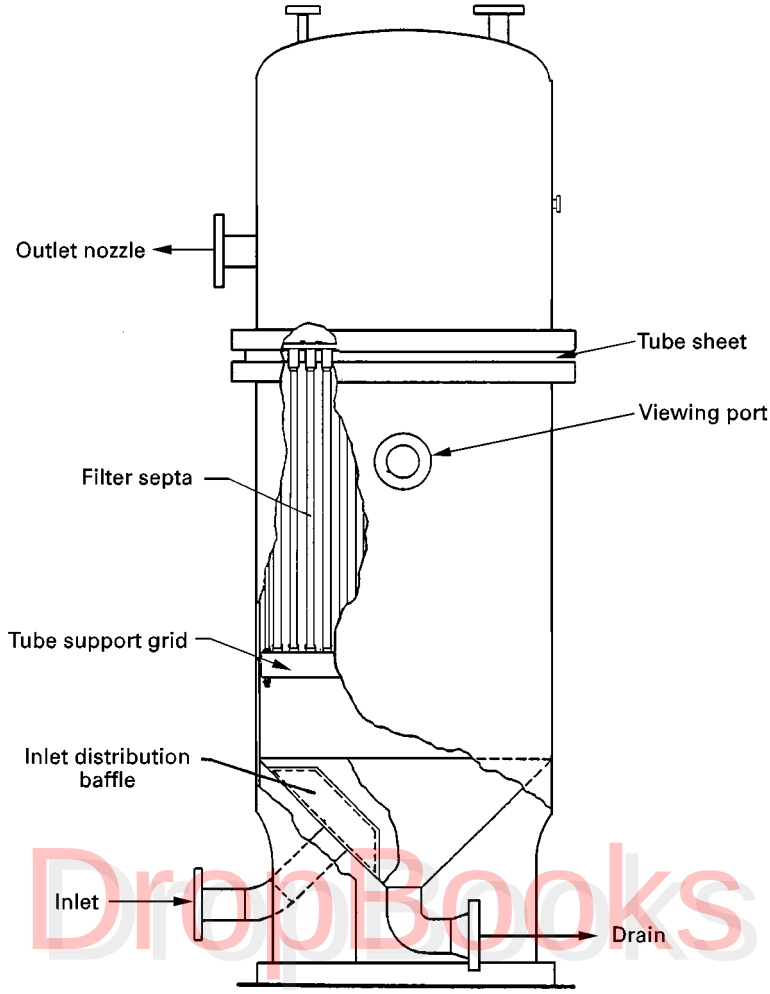

Figure 4 Pre-coat system design: top tube sheet.

in a variety of diameters and lengths; large vessels accommodate 127–203 cm elements. A variety of fibres, including polyester, polypropylene, nylon and carbon, are used for the yarn winding, depending on the application.

It should be noted that powdered resin pre-coats can be applied to traditional septa such as leaf filters or plate and frame filter presses. Resin pre-coats can also be used in beds in columns similar to bead resins. This type of usage is typically limited to small bed depths due to hydraulic considerations (pressure drop across the bed).

Operation

The slurry tank is filled with water, preferably deionized. Individual powdered components or a premixture of the same are weighed out, added to the water and mixed using a mechanical stirrer. Generally, dosage rates of the pre-coat vary from 0.73 to 1.46 dry kg pre-coat per square metre of surface area on the septum. This dosage will result in a pre-coat of 0.63–1.27 cm depth. Recently, thin-layer pre-coats have become popular with dosage rates of 0.24–0.49 dry kg m^{-2}. Overlays and underlays are often pre-coated at even lower dosage rates of 0.10–0.24 dry kg m^{-2}.

The pre-coat is applied to the filter or septum as a slurry via the pre-coat pump at an area flow rate of 0.68–1.70 L s^{-1} m^{-2}. The slurry concentration as it enters the pre-coat vessel is critical. A concentration of 0.05% is ideal, though not always attainable. The pre-coat recycle loop shown in Figure 3 is used to optimize slurry concentration entering the pre-coat vessel through dilution. The objective is to lay down a uniform pre-coat across the length of each and every filter element. A low slurry concentration will translate into small floc size. The small floc, in turn, can be carried to the upper portion of the vessel despite low flow in that region. Alternatively, larger floc particles

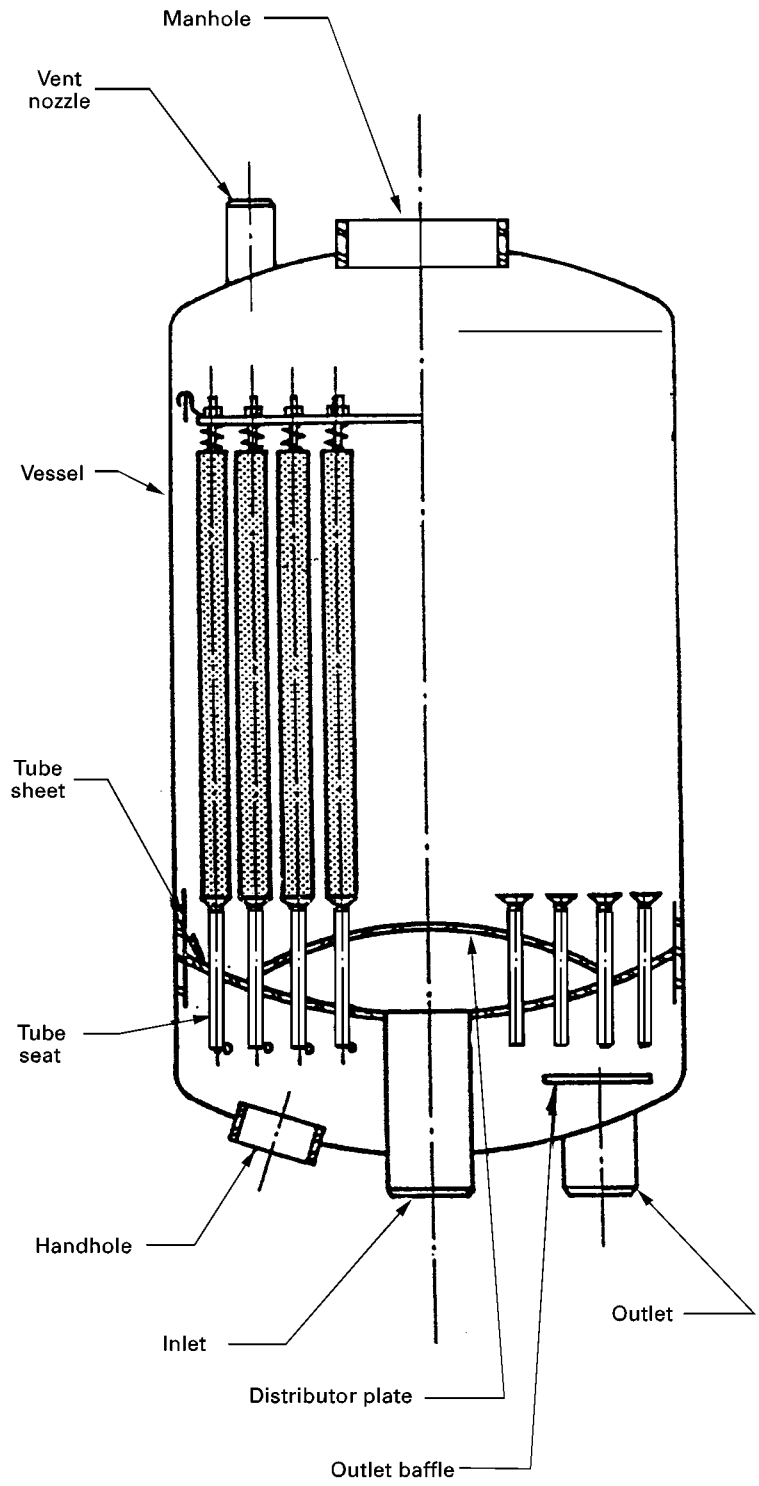

Figure 5 Pre-coat system design: bottom tube sheet.

can reach a point where the flow carrying them upward is balanced by gravity. The consequence of this condition is reflocculation where floc grows in size, then falls towards the bottom. Even if the refloc eventually pre-coats the filter, its large particle size creates an uneven surface. Pre-coating, along with recirculation, continues until all powdered material is deposited on the surface of the filter elements. This process takes 10–90 min, after which additional recirculation allows the system to clear (residual turbid-

ity in water dissipates). At this point, the pre-coat is ready for standby or service. If the standby option is chosen, the hold pump maintains sufficient flow across the pre-coat to prevent it from falling off the filter element. In some instances, application of additional pre-coat material – a technique known as body feed – during the service cycle greatly enhances system performance.

During the service cycle, the pre-coat removes contaminants from the feed solution via ion exchange, adsorption and filtration mechanisms. The purified solution passes through the pre-coat and the filter element and emerges as product. Water quality, usually measured by conductivity and/or ionic content, and pressure drop across the pre-coat are monitored throughout the service cycle. During the service cycle, the pre-coat contracts and tightens as it is loaded with ions, organics and colloids. The area flow rate during the service cycle is 2.0–2.7 L s^{-1} m^{-2}. Deterioration of one of the measurement parameters determines the end point of the service cycle (e.g. differential pressure drop across the pre-coat increases by 0.70 kg cm^{-2}. At this point, the expended pre-coat is removed from the filter via back-wash. During back-washing flow across the pre-coat is reversed and the solids are slurried to waste. With the evolution of pre-coat technology, back-washing has also changed from simple surges of water or air to combinations of high pressure air and water. The scrubbing properties of the air and water combination are further enhanced by repeated applications, each with a different level of water in the pre-coat vessel. The greatest effect occurs on the element in the immediate vicinity of the air–water interface in the vessel. These elaborate processes are designed to remove pre-coat particles embedded in the filter element as well as the bulk pre-coat on the surface. At the conclusion of the back-washing steps, the system is ready for the next pre-coat.

Over the years, mechanical innovations have improved upon the original pre-coating process. Metering tanks feed the resin slurry into the mix tank at a specific rate, thus offering excellent control over slurry concentration. Draft tubes installed in the pre-coat vessel carry pre-coat particles directly from the inlet at the bottom of the vessel to the top of the vessel. With the draft tube design, slurry flow is split and the filter elements are exposed to pre-coat flow both top-down and bottom-up. The result is a more even pre-coat deposition on the element.

Applications

Powdered pre-coats have been useful in a wide variety of applications. Among the most common are polishing steam condensates; treatment of low activity radioactive wastes; decolorization, decalcification and clarification of sugar juices, sugar syrups and polyhydric alcohols; clarification of fluids containing electrolytes and colloids; purification of antibiotics, vitamins and other pharmaceuticals; removal of toxic and noxious organics from potable and industrial waters; recovery of precious metals; pretreatment of high purity water systems; recovery of catalyst residues from reaction mixtures; and removal of traces of acids, bases and salts from polar solvents. Most pre-coat applications involve removal of impurities present in low concentrations.

Powdered ion exchange resin pre-coats were first utilized in electric power plants. Originally, bead-form ion exchange resins were used to remove (polish) soluble ionic contaminants from condensate prior to recycle to the steam generators. Powdered resin pre-coats combined this ion exchange polishing capability with filtration of colloidal and particulate materials such as a mixture of transition metal oxides (Fe, Cu, Co, etc.) present in steam condensate due to erosion or corrosion. Pre-coat technology was applied in both nuclear and fossil steam-generating plants. This technology proved particularly valuable in the boiling water reactor (BWR)-type nuclear plants. Unlike their bead counterparts, powdered pre-coats generate no liquid waste regenerants since they are designed to be nonregenerable and disposable solids. Issues such as short run lengths due to the high level of suspended solids in BWR condensate led to the development of pre-coats incorporating fibre in addition to powdered resins. The use of powdered resins in nuclear condensate polishing is so prevalent that operational guidelines have been issued by the US industry (see Further Reading).

In addition to condensate polishing, powdered pre-coats are used in reactor water clean-up (RWCU), elevated temperature heater drain and fuel pool purification applications in nuclear power plants. Powdered pre-coats have also been widely adopted in nuclear power plants for radioactive waste systems. In these systems, the powdered resin materials are pre-coated directly on to existing filters replacing diatomaceous earth or cellulose fibres. They offer both efficient ion exchange capacity and corrosion product filtration. These pre-coat materials have been tailored to remove specific troublesome radionuclides with a long half-life such as ^{60}Co, ^{90}Sr, ^{134}Cs and ^{137}Cs. In addition to synthetic organic ion exchange resins, both natural and synthetic zeolites are utilized in selective radioactive waste applications. Similarly, alternative resin types such as ammonium form or morpholinium form strongly acidic cation and weakly acidic cation components

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are commercially available to customers from public utilities.

Blaine and Down first described the application of powdered ion exchange resin technology for decolorizing sugar syrups. Colour bodies in sugar syrups are high molecular weight entities which anion exchange resins typically selectively remove. Blaine and Down reasoned that reaction kinetics, as controlled by particle diffusion, are much slower in viscous liquids such as sugar syrups than typically found in water applications. Consequently, finely divided powdered resins decolorize much more rapidly than their larger bead counterparts. Moreover, the powdered resins are used as disposable, non-regenerable filter pre-coats, thus eliminating the need for chemical regenerants for the ion exchange resin. These pre-coats offer increased operating capacity for colour removal without generating unacceptably high pressure drop across the pre-coat. The high decolorization capacities also result in lowered sugar losses from dilution during the sweetening-off and sweetening-on steps. Sugar pre-coats offer clarification through filtration in addition to decolorization.

The initial sugar decolorization pre-coats contained mixtures of anion and cation exchange resins. Over the years, commercial formulations have expanded to include mixtures of selective anion exchange resins, powdered activated carbons and/or chopped fibres depending on the specific application. These formulations are tailored for the clarification and decolorization of sugar syrups (cane, corn and beet), fruit juices (grape, apple) and polyhydric alcohols (glycerine, sorbitol). The decolorization performance of these pre-coats compares favourably with traditional technologies (bone char, granulated activated carbon and regenerable ion exchange resins), especially as a final polishing step. In addition, tailored pre-coats remove species that cause undesirable taste and odour in the product. Pre-coats also offer limited de-ashing (deionizing) capacity in sugar syrups. De-ashing is limited by the number of ion exchange sites, not to mention the competition from colour bodies for the anion exchange sites. Low capital investment, limited space requirements and reduced energy costs are cited as advantages for the pre-coat technology in sugar applications.

Future Directions

Powdered pre-coat usage has been declining in recent years, especially in utility applications. Few new power generation plants are designed and built with pre-coat systems. In nuclear power plants, the cost of radioactive resin disposal continues to rise dramatically. Thus, utilities have minimized the use of ion exchange resins, including powdered pre-coats, in these facilities. The introduction of thin-layer pre-coat technology has further accelerated this trend. Because of the risk of leaching after burial, concerns are increasing over the ultimate fate of formulations containing biodegradable fibres such as cellulose. Fortunately, alternative fibres such as PAN, nylon, polyethylene and polyester fibres can be substituted for the cellulosic fibre in pre-coat formulations. The PAN fibre formulations offer the added advantage of extended run length to a given differential pressure drop end point. Pleated filter elements have replaced yarn wound filter elements in many plants. The pleated elements offer excellent iron removal without using a powdered pre-coat.

Premix pre-coats are increasingly popular, replacing traditional powdered component pre-coats. Premixes offer the convenience of a one-step operation and do not necessitate operator adjustments during pre-coating. Of course, the flexibility of adjusting powdered component ratios on each cycle depending on influent conditions is lost with premixes. As available human resources (operators) shrink, the popularity of premixes will continue to grow. Customers demand and receive ultrahigh purity powdered pre-coats. Specification limits on residual contaminants such as iron, copper, aluminium, silica, sodium, potassium, calcium and magnesium for powdered pre-coats are typically set at 50 p.p.m., 25 p.p.m. or even 10 p.p.m. by dry weight. These stringent specifications apply to both resin components and pre-coat formulations. Increasingly, selective ion exchange media, including zeolites, are being incorporated into pre-coat formulations. Radioactive waste applications, discussed above, are a perfect example of this trend. Custom-designed powdered pre-coats of all types are growing in popularity.

See also: **II/Ion Exchange:** Historical Development; Organic Ion Exchangers; Theory of Ion Exchange.

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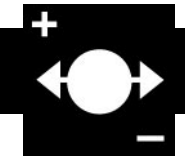
PREPARATIVE ELECTROPHORESIS



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Introduction

The development of electrophoresis in the early part of the 20th century proved to be an extremely important tool for the separation of biologically important molecules such as amino acids, peptides, proteins and DNA. There are four main types of electrophoresis, known as zone electrophoresis, step-field electrophoresis, isoelectric focusing and isotachophoresis. This review discusses the development of zone electrophoresis and step-field electrophoresis for preparative applications.

Electrophoresis instruments use mobility differences in the presence of an electric field to separate mixtures into individual components. The mobility differences between individual species are proportional to the net charge to size ratios of the species. Therefore, separations are most effective for solutes with large differences in this ratio. Additional separation mechanisms, such as molecular sieving using gels, have since been incorporated in the modes of electrophoresis to increase the range of applicability. At present, there are a wide range of electrophoresis instruments and methods which are used routinely in the biotechnology industry for analytical measurements. In contrast, the use of preparative electrophoresis is far less extensive.

The major distinction between analytical and preparative electrophoresis lies in the size and processing of samples. In the case of preparative electrophoresis, sample sizes are generally much larger (mg to g) in comparison with analytical electrophoresis (ng to μg) depending on the availability of the species of interest. Such large samples must not be too crude because irreversible adsorption of some unwanted species can render the system inoperable. The samples also require the collection of fractions after separation, which is often not the case with analytical separations. Thus, after separation, it is essential that the solutes can be easily removed from the buffer solution if they are to be prepared as pure compounds. The constitutions of buffer solutions must therefore be carefully chosen, preferably with volatile components, which facilitate collection of the species of interest.

Preparative electrophoresis systems require scaling up from the respective analytical systems and modifications to the instrumentation have been made which attempt to contend with complications arising from the scale-up. For instance, an inherent problem with electrophoresis is thermal convection caused by the flow of ions in the presence of an applied electric field. As electrophoresis systems are scaled up for preparative applications, convection problems and heat dissipation in the system become more significant owing to the decrease in the relative surface area. Anticonvective media such as filter paper, agarose, starch, glass powder or polyacrylamide have been used to limit these convection processes. The use of anticonvective media, however, has led to other problems, such as adsorption, endosmosis and diffusion. These combined factors have therefore prompted the development of a number of different designs of preparative electrophoresis systems that are suitable for continuous or batch-wise separations of multicomponent mixtures. Two basic strategies have evolved which are collectively termed preparative free-flowing electrophoresis or preparative gel electrophoresis. Both strategies exploit the same basic electrophoresis process for the separation, although both approaches have been used in a variety of configurations.

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